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Japanese Published Unexamined Patent Application (A) No. 57-092502, published June 9, 1982; Application Filing No. 55-166732, filed November 28, 1980; Inventor(s): Yoshio Oda et al.; Assignee: Asahi Glass Corporation; Japanese Title: Method to Store or Transport Hydrofluoric Acid

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## METHOD TO STORE OR TRANSPORT HYDROFLUORIC ACID

### CLAIM(S)

1) A method for storing or transporting hydrofluoric acid characterized by the following steps: a  $P_y-(HF)_n$  complex ( $P_y$  = pyridine,  $n \geq 3$ ) produced by reacting pyridine to hydrofluoric acid or a  $P_y-(HF)_m$  complex ( $m \geq 8$ ) produced by reacting a  $P_y-HCl$  complex to 8 equivalent amount or more of hydrofluoric acid is stored or transported; at the prescribed time and place, said  $P_y-(HF)_n$  or  $P_y-(HF)_m$  is distilled to dissociate  $(n-3)HF$  or  $(m-3)HF$  from  $P_y-(HF)_2$ .

2) A method for transporting hydrofluoric acid, as cited in Claim 1, characterized in that  $P_y-(HF)_n$  or  $P_y-(HF)_m$  is produced by adding the  $P_y-(HF)_2$  to hydrofluoric acid.

3) A method for transporting hydrofluoric acid, characterized by the following steps: a  $P_y-(HF)_n$  complex ( $P_y$  = pyridine,  $n \geq 3$ ) produced by reacting pyridine to hydrofluoric acid or a  $P_y-(HF)_m$  complex ( $m \geq 8$ )

produced by reacting a  $P_y\text{-HCl}$  complex to 8 equivalent amount or more of hydrofluoric acid is stored or transported, and at the prescribed time and place, said  $P_y\text{-(HF)}_n$  or  $P_y\text{-(HF)}_m$  is distilled to dissociate  $(n-3)\text{HF}$  or  $(m-3)\text{HF}$ ; hydrochloric acid is added to this  $P_y\text{-(HF)}_2$ , and this product is distilled to dissociate  $3\text{HF}$  from  $P_y\text{-HCl}$ .

4) A method for storing or transporting hydrofluoric acid, as cited in Claim 3, wherein ammonia is reacted to  $P_y\text{-HCl}$ , and the produced  $P_y$  is used as a material for producing the  $P_y\text{-(HF)}_n$ .

5) A method for storing or transporting hydrofluoric acid, as cited in Claim 3, wherein the  $P_y\text{-HCl}$  is used as a material for producing  $P_y\text{-(HF)}_m$ .

6) A method for storing or transporting hydrofluoric acid, as cited in Claim 3, wherein at least 8 equivalent amount or more of hydrofluoric acid is reacted to the  $P_y\text{-HCl}$  in a solvent of 1, 1, 2-trichloro-1, 2, 2-trifluoroethane.

## DETAILED DESCRIPTION OF THE INVENTION

The present invention pertains to a method to safely store or transport hydrofluoric acid.

Hydrofluoric acid is extremely corrosive, and its corrosiveness is vitally increased if it is exposed to even a slight amount of water. Since it has a boiling point  $20^\circ\text{C}$ , it has pressure higher than atmospheric pressure.

Therefore, it is generally filled in an anti-pressure container made of steel for storing and transporting.

With this prior art method, however, various complex safety measures are needed at a time of using it, which is a problem.

Therefore, the inventors of the present invention made an effort to find an easy storing or transporting method for hydrofluoric acid that is really safe and requires low cost, and produced the present invention.

The present invention presents a method for storing or transporting hydrofluoric acid characterized by the following: a  $P_y-(HF)_n$  complex ( $P_y$  = pyridine,  $n \geq 3$ ) produced by reacting pyridine to hydrofluoric acid or a  $P_y-(HF)_m$  complex ( $m \geq 8$ ) produced by reacting a  $P_y-HCl$  complex to 8 equivalent amount or more of hydrofluoric acid is stored or transported; at the prescribed time and place, said  $P_y-(HF)_n$  or  $P_y-(HF)_m$  is distilled to dissociate  $(n-3)HF$  or  $(m-3)HF$  from  $P_y-(HF)_2$ . The present invention also presents a method to store or transport hydrofluoric acid characterized in that a  $P_y-(HF)_n$  complex ( $P_y$  = pyridine,  $n \geq 3$ ) produced by reacting pyridine to hydrofluoric acid or a  $P_y-(HF)_m$  complex ( $m \geq 8$ ) produced by reacting a  $P_y-HCl$  complex to 8 equivalent amount or more of hydrofluoric acid is stored or transported, and at the prescribed time and place, said  $P_y-(HF)_n$  or  $P_y-(HF)_m$  is distilled to dissociate  $(n-3)HF$  or  $(m-3)HF$  from  $P_y-(HF)_2$ , and in that after

hydrochloric acid is added to this  $P_y-(HF)_2$ , this product is distilled to dissociate 3HF from  $P_y-HCl$ .

More specifically, the pyridine-hydrofluoric acid complex,  $P_y-(Hf)_n$ , is a stable compound without a corrosive property. In this compound, a variable amount of HF can be put into a specific amount of pyridine to form the pyridine-hydrofluoric complex. Therefore, if said complex can be produced from a large amount of n and a specific amount of pyridine, a high volume of hydrofluoric acid can be stored or transported. The pyridine-hydrofluoric acid complex is a publicly known substance, but using this as a method for storing or transporting hydrofluoric acid is totally a novelty method, so the method explained below is an unprecedented method.

The pyridine-hydrofluoric acid complex containing the prescribed amount of HF will not be useful as the means of storing and transporting the hydrofluoric acid if hydrofluoric acid cannot be extracted from it.

Regarding the extraction of hydrofluoric acid from the pyridine-hydrofluoric acid complex, the inventors found an excellent method, and therefore they could implement the method for storing or transporting hydrofluoric acid.

The amount of hydrofluoric acid per 1 mol of pyridine in the complex can be properly selected, but it is preferable to use at least 3 mol or 8 mol of

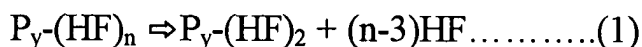
hydrofluoric acid to raise the use rate of pyridine (e.g.,  $n$  or  $m \geq 3$ , preferably,  $n$  or  $m \geq 8$ ). The maximum limit needs not be specified, but  $n$  or  $m$  is preferably less than 20, more preferably, 15 or less.

The pyridine-hydrofluoric acid complex thus produced is can be easily produced by blowing hydrofluoric acid into pyridine or pyridine-hydrochloric acid complex.

The pyridine-hydrofluoric acid complex thus produced is a highly stable substance, for the hydrofluoric acid steam pressure being low, therefore, can be stored for the desired length of time. Even if a generally used container is used for it, for example, a polyethylene container, it can be transported to anywhere.

Then, at the prescribed time and place, the hydrofluoric acid can be extracted easily as follows.

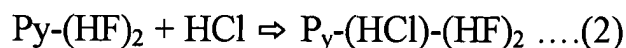
More specifically, when the pyridine-hydrofluoric acid complex is distilled in vacuum, most of the hydrofluoric acid can be dissociated according to the reaction indicated below:



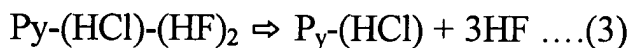
Even if about 3 equivalent amount of hydrofluoric acid remains in the case when the value of  $n$  is high, the use rate of the pyridine is sufficiently

high enough to return the  $\text{Py}-(\text{HF})_2$  to the initial position to be used as a raw material for producing the  $\text{Py}-(\text{HF})_2$ .

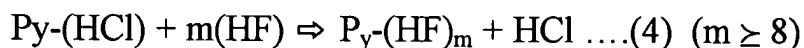
The inventors, further, have recognized that it is necessary to dissociate this 3 equivalent amount of hydrofluoric acid to improve the use rate of pyridine, and found the following easy and excellent means. More specifically hydrochloric acid is reacted to the  $\text{Py}-(\text{HF})_2$ , and the pyridine-hydrochloric acid-hydrofluoric acid complex,  $\text{Py}-(\text{HCl})-(\text{HF})_2$ , is produced, according to:



By distilling this complex in vacuum, 3 equivalent amount of hydrofluoric acid is easily dissociated, and the pyridine-hydrochloric acid complex,  $\text{Py}-(\text{HCl})$  is produced, as indicated below:



This pyridine-hydrofluoric complex is returned to the initial position and reacted to ammonia to reproduce pyridine or reacted to 8 equivalent amount of hydrofluoric acid, as indicated below, to reproduce  $\text{Py}-(\text{HF})_m$ :



The pyridine thus reproduced is used again to produce the pyridine-hydrofluoric acid complex by reacting it to hydrofluoric acid. The produced

Py-(HF)<sub>m</sub> can be used for storing or transporting the hydrofluoric acid, or more hydrofluoric acid can be added to provide it with the higher value of m.

As to the means of producing the P<sub>y</sub>-(HF)<sub>m</sub> by reacting 8 or more equivalent amount of hydrofluoric acid to the aforementioned P<sub>y</sub>-(HCl), it is preferred to conduct this reaction in a solvent of 1,1,2 – trichloro-1,2,2 trifluoroethane. The reason for this is that when the reaction is conducted in the aforementioned solvent, the produced P<sub>y</sub>-(HF)<sub>m</sub> (m ≥ 8) is insoluble in the aforementioned solvent, while on the other hand the dissociated hydrochloric acid is soluble, so the P<sub>y</sub>-(HF)<sub>m</sub> and HCl are automatically dissociated, which is convenient.

In the above explanation, the pyridine - hydrofluoric acid complex was explained, but instead of pyridine, an organic amine, e.g., picoline, quinoline, trimethylamine, or triethylamine, can be likewise used.

(Embodiment Example 1)

A hydrofluorine gas 11 mol was put in a container containing pyridine 1 mol at a room temperature. Py-(HF)<sub>11</sub> was produced by the amount of 299 g. This product was put in a polyethylene bottle and the bottle was plugged. After 200 days of storing the product, it was removed from the bottle, and distilled by 5 mmHg at 150°C. The dissociated hydrofluoric acid was 8 mol. Subsequently, to dissociate the HF from the remaining Py-(HF)<sub>2</sub>, it was

reacted to 1 mol of hydrochloric acid, and 1 mol of  $\text{Py}-(\text{HCl})-(\text{HF})_2$  was produced. Subsequently, this was distilled at 5 mmHg and  $140^\circ\text{C}$ , and 1 mol of  $\text{Py}-\text{HCl}$  and 3 mol of hydrofluoric acid were produced.

To this 1 mol of  $\text{Py}-\text{HCl}$ , 1 mol of ammonia was reacted at a room temperature, and pyridine was reproduced. As explained above, there was 11 mol of dissociated hydrofluoric acid, which was the same amount as that initially stored, so there was no loss at all. Also, the above operation was very simple.

(Embodiment Example 2)

The  $\text{Py}-\text{HCl}$  1 mol produced by the same operation as in Embodiment example 1 was put in a container containing 1000 ml of 1,1,2-trichloro-1,2,2-trifluoroethane to produce the suspension.

Subsequently, 14 mol of hydrofluoric acid was poured into this suspension to react to  $\text{Py}-\text{HCl}$ . The reaction product was clearly divided into 2 phases, and the top layer was the  $\text{Py}-(\text{HF})_{14}$ , while the bottom layer was 1,1,2 – trichloro-1,2,2 – trifluoroethane in which hydrochloric acid was dissolved.

(Embodiment Example 3)



Instead of pyridine of Embodiment Example 1, triethylamine was used. The result indicated, as with the case of Embodiment Example 1, that there was no loss of hydrofluoric acid and its 100% was retained.

(Embodiment Example 4)

Instead of pyridine of Embodiment Example 1, quinoline was used. The result showed, as with the case of Embodiment Example 2, the quinoline-hydrofluorine complex was separated as the top layer over the solvent of 1,1,2-trichloro-1,2,2-trifluoroethane.

(Embodiment Example 5)

The Py-(HF) by 15 mol produced in the same manner as in Embodiment Example 1 was put in a polyethylene bottle and the bottle was plugged. This product was transported by a vehicle by 100 Km. Then, as with the case of Embodiment Example 1, hydrofluoric acid was dissociated. It was found that there was no loss of hydrofluoric acid.

Translations  
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